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First examples of a covalent bond between gold and selenophene

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Experimental Section

^{1}H, ^{13}C, and ^{77}Se NMR spectra were recorded on a Bruker Avance Neo spectrometer at 400.0, 100, and 76 MHz, respectively, at 303 K in DMSO-d_6 solution. The ^{1}H chemical shifts are given relative to DMSO, and ^{77}Se relative to dimethyl selenide. Infrared (IR) spectra were recorded with a Prestige-21 FTIR spectrometer (Shimadzu, Kyoto, Japan). Single crystals of were investigated on a Rigaku, XtaLAB Synergy, Dualflex, HyPix diffractometer.

A solution of compound **1** or **4** (0.128 g, 0.497 mmol) dissolved in MeCN (8 ml) was added to $NaAuCl_4$ (0.150 g, 0.414 mmol) dissolved in water (10 ml). Orange precipitates formed as the reaction was stirred at room temperature for 3 h. It was collected by filtration, rinsed with water (10 ml), and dried under reduced pressure. The orange solid was obtained.

[2-(Benzo[*b*]selenophen-2-yl)pyridin-1-ium-1-yl]trichloroaurate (**2**). Yield: 86%. mp=173-175 °C (dec.) ^{1}H NMR (DMSO-d_6, 400 MHz): δ = 8.58 (ddd, J= 4.9; 1.8; 1.0 *Hz*, 1H), 8.34 (s, 1H), 8.11 (dt, *J*= 8.0; 1.1 Hz, 1H), 8.05-8.02 (m, 1H), 7.92-7.85 (m, 2H), 7.41-7.28 (m, 3H). ^{13}C NMR (101 MHz, hot DMSO-d_6) δ = 153.0; 149.6; 148.8; 143.2; 141.3; 137.2; 126.0; 125.9; 125.3; 124.9; 124.8; 123.2; 118.7. ^{77}Se NMR ($CDCl_3$, 76 MHz): δ = 525.4.

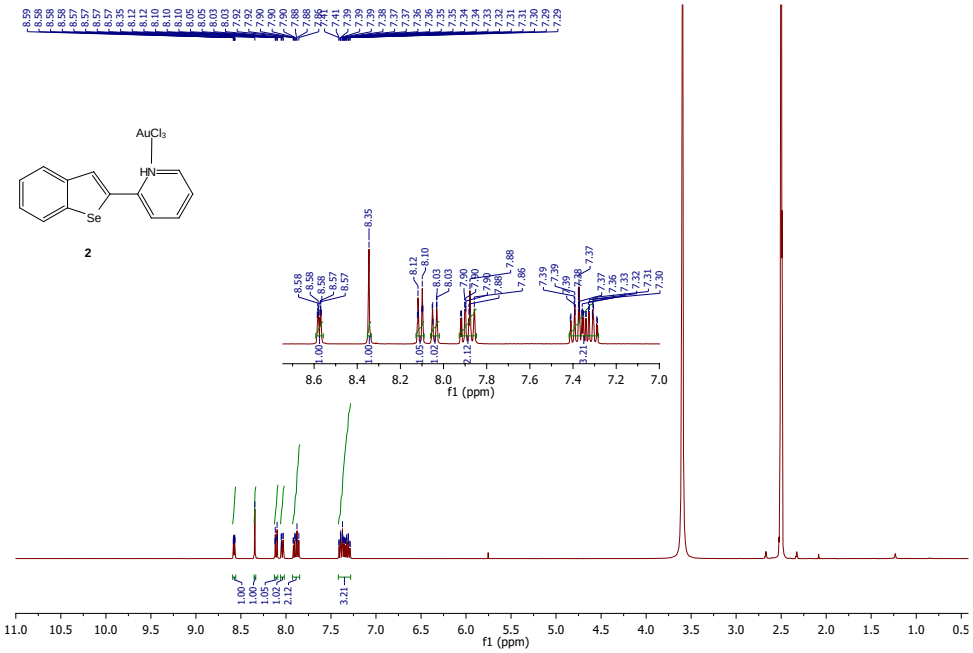
[2-(Benzo[*b*]selenophen-3-yl)pyridin-1-ium-1-yl]trichloroaurate (**5**). ^{1}H NMR (DMSO-d_6, 400 MHz): δ = 9.46-9.42 (m, 1H), 9.22-9.17 (m, 1H), 8.35-8.28 (m, 1H), 8.25-8.20 (m, 1H), 8.12-8.08 (m, 1H), 7.67-7.61 (m, 1H), 7.47-7.41 (m, 2H). ^{13}C NMR (101 MHz, hot DMSO-d_6) δ = 146.5, 142.0, 138.7, 134.4, 126.4, 125.2, 125.0, 124.94, 124.87, 123.7. ^{77}Se NMR ($CDCl_3$, 76 MHz): δ = 599.6.

A suspension of betain **2** or **5** in MeCN (5 ml) was refluxed for 5 h. After cooling to room temperature, a bright yellow solid formed rapidly. The product was filtered off and dried.

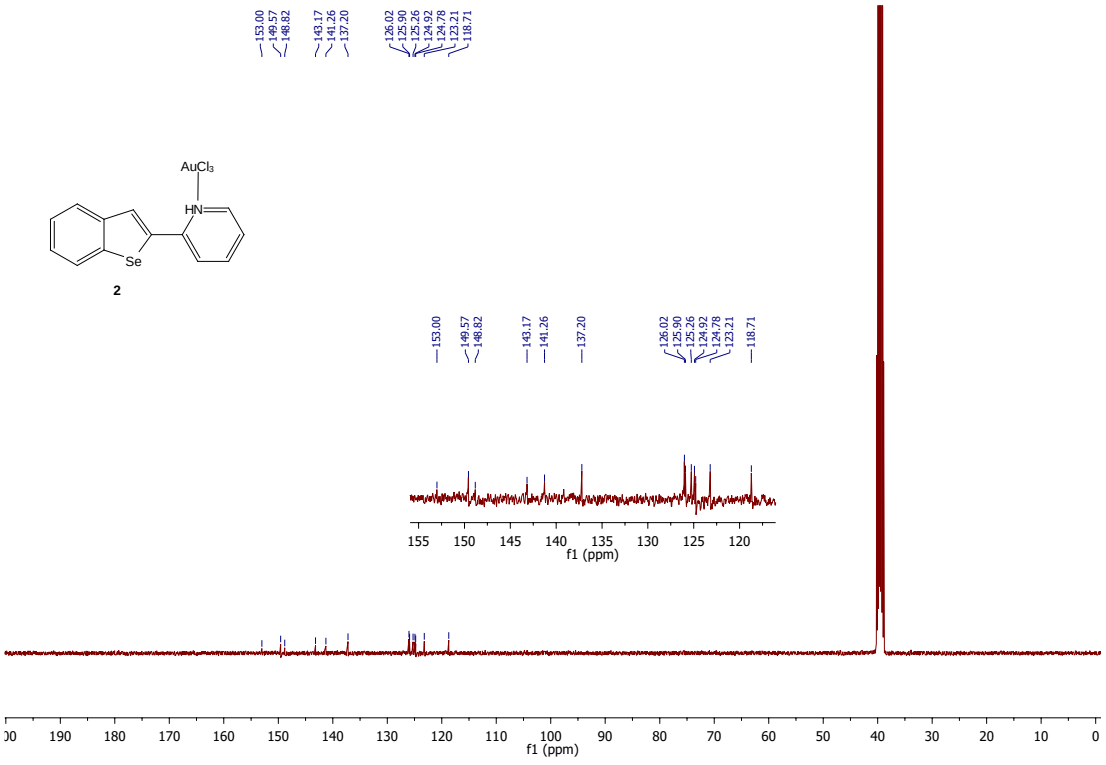
[2-(Pyridin-2-yl)benzo[*b*]selenophen-3-yl]gold(III) chloride (**3**). Yield: 36%. mp=195-198 °C (dec.). IR_{KBr}: υ, 3545, 3483, 3412, 1638, 1617, 1476, 668 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ = 8.83-8.81(m, 1H), 8.78 (s, 1H), 8.27-8.19 (m, 3H), 8.03-8.01 (m, 1H), 7.71-7.68 (m, 1H), 7.49-7.40 (m, 2H). Elem. analysis: calcd. for $C_{13}H_8AuCl_2NSe$: C, 29.74; H, 1.54; N, 2.67; found: C, 29.51; H, 1.56; N, 2.49.

[3-(Pyridin-2-yl)benzo[*b*]selenophen-2-yl]gold(III) chloride (**6**). Yield: 28%. mp>250 °C. IR_{KBr}: υ, 3556, 3486, 3412, 1600, 1485, 1476, 1412, 991, 774 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz): δ = 9.36-9.34 (m, 1H), 8.47-8.34 (m, 3H), 8.15-8.13 (m, 1H), 7.71-7.66 (m, 1H), 7.52-7.48 (m, 1H), 7.44-7.39 (m, 1H). Elem. analysis: calcd. for $C_{13}H_8AuCl_2NSe$: C, 29.74; H, 1.54; N, 2.67; found: C, 29.44; H, 1.45; N, 2.36.

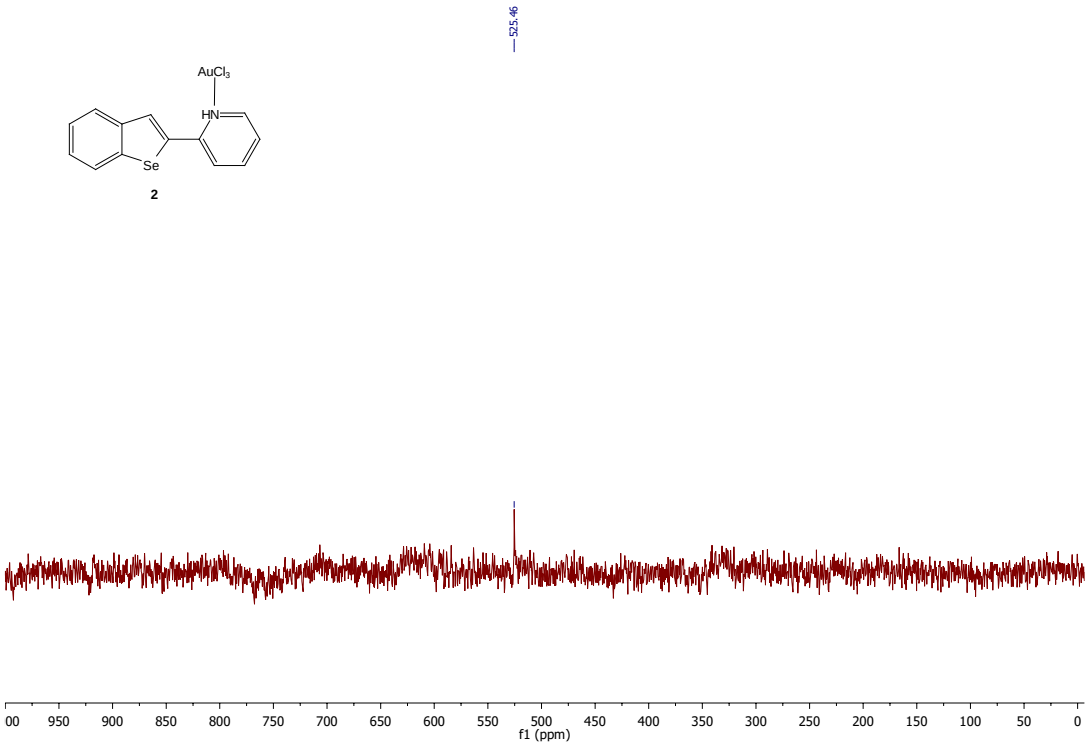
^{1}H NMR spectrum of **2**



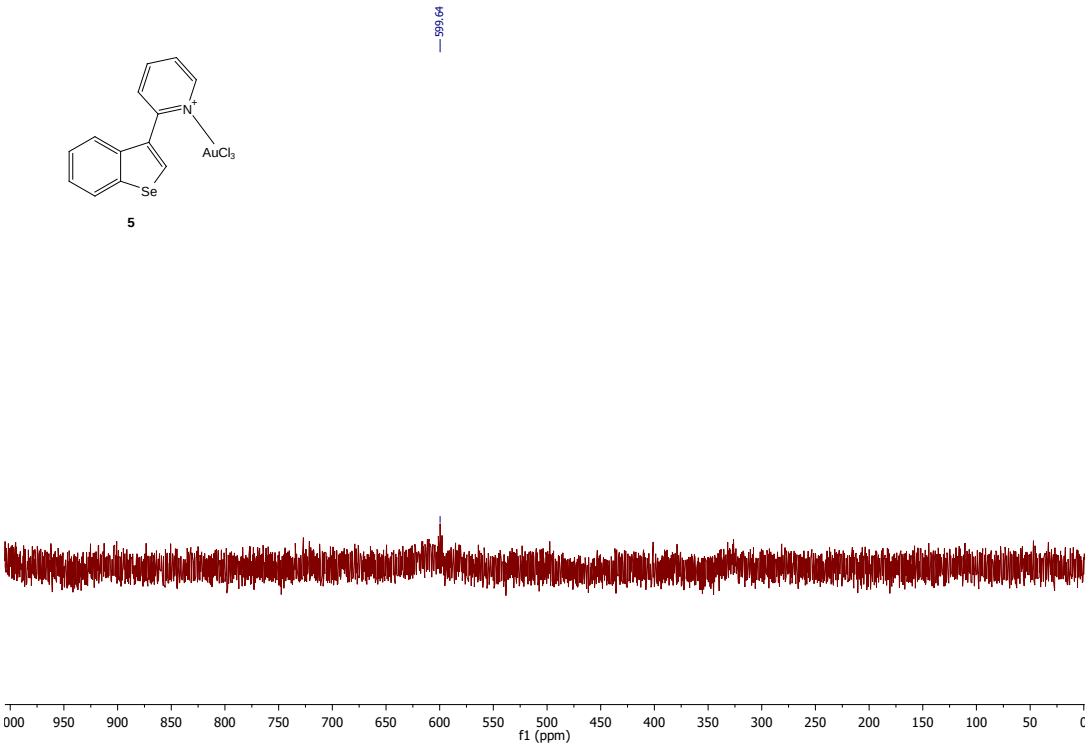
^{13}C NMR spectrum of **2**



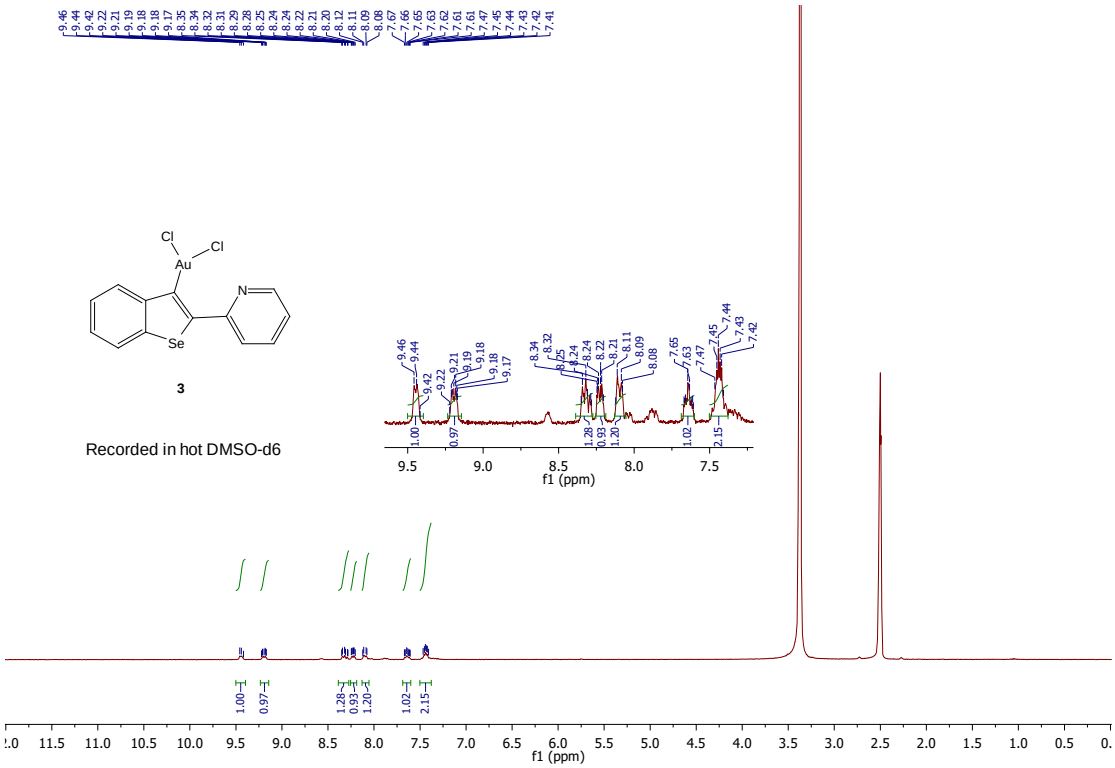
^{77}Se NMR spectrum of **2**



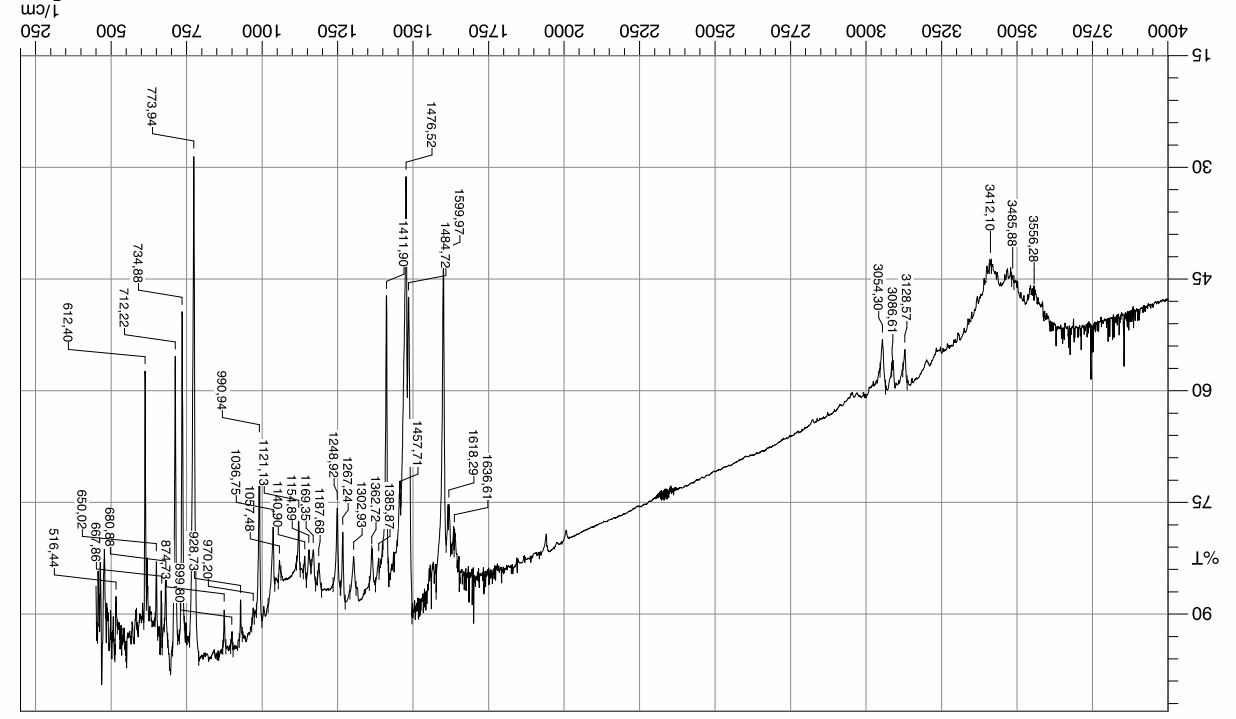
^{77}Se NMR spectrum of **5**



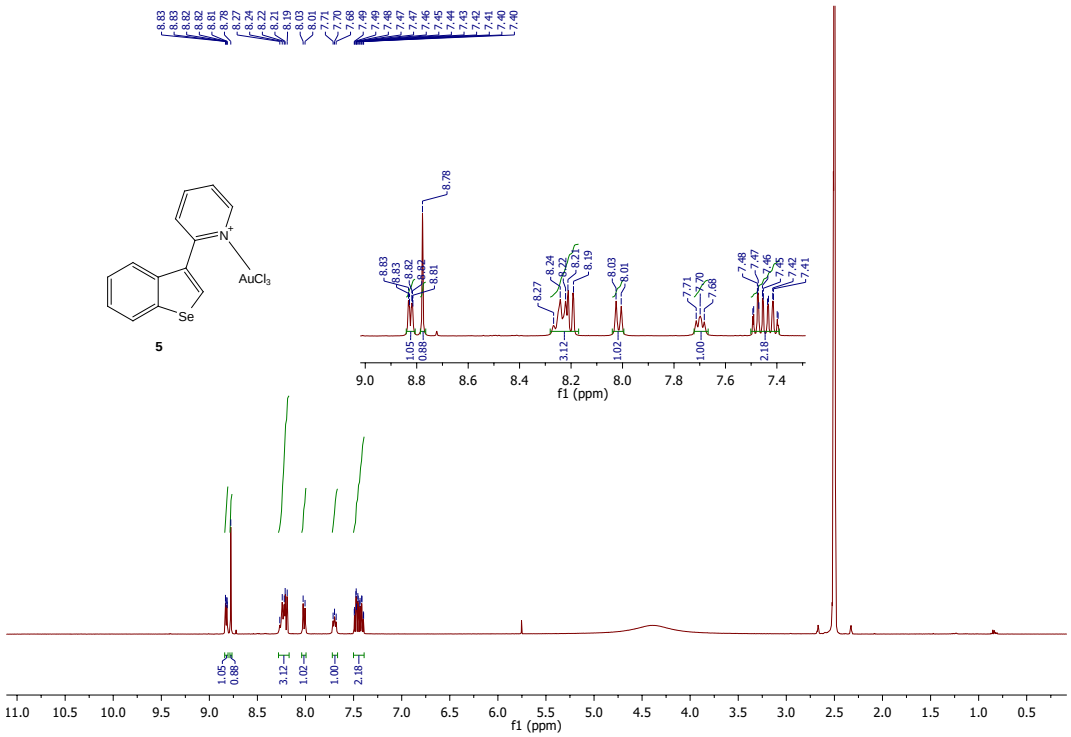
^{13}C NMR spectrum of **3**



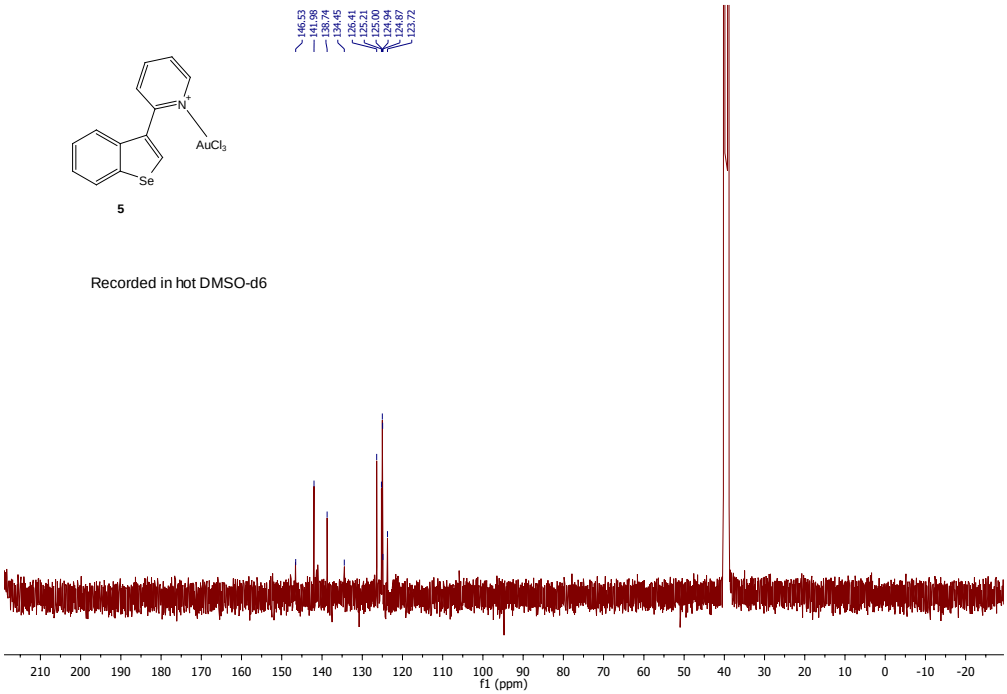
IR spectrum of **3**



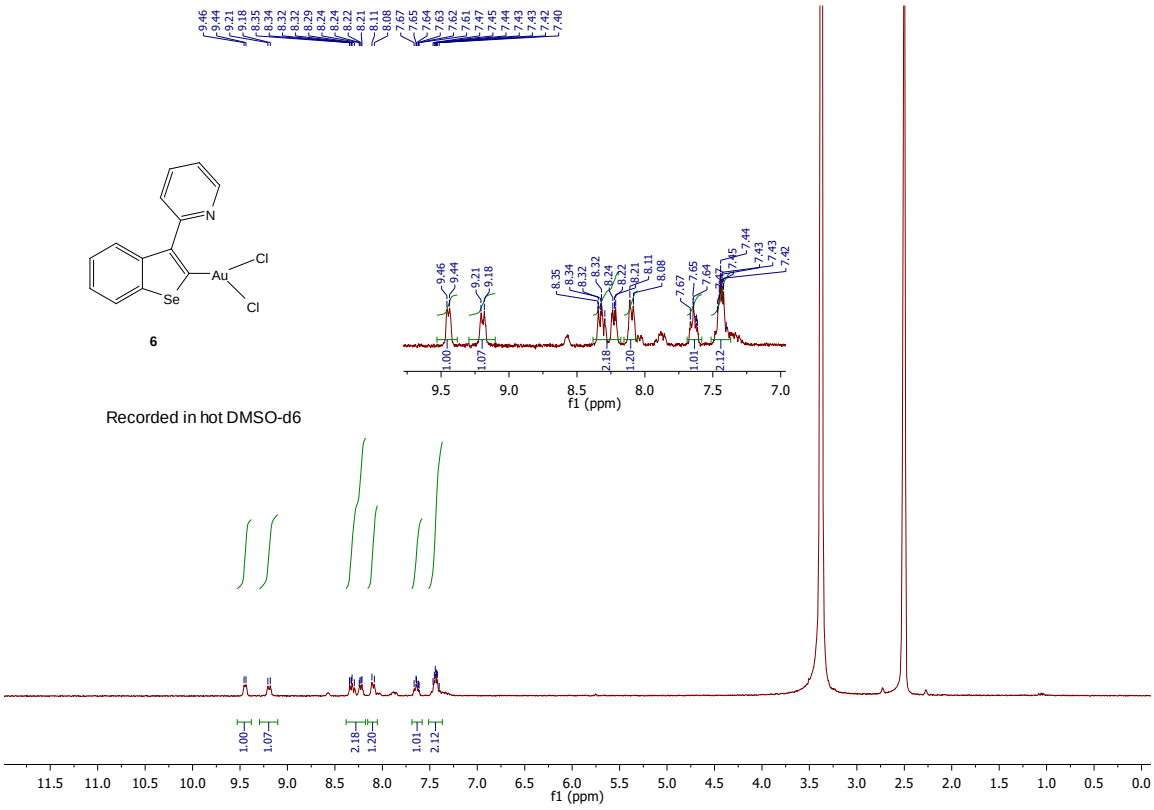
^{1}H NMR spectrum of **5**



^{113}C NMR spectrum of **5**



^{1}H NMR spectrum of **6**



IR spectrum of **6**